

Pectin Extract Optimization from Ripe Cocoa Pod Husks using Response Surface Methodology and Artificial Neural Network

To Cite:

Ekperi NI, Achinike W. Pectin Extract Optimization from Ripe Cocoa Pod Husks using Response Surface Methodology and Artificial Neural Network. *Indian Journal of Engineering*, 2022, 19(52), 403-409

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Peer-Review History

Received: 27 July 2022

Reviewed & Revised: 29/July/2022 to 09/September/2022

Accepted: 11 September 2022

Published: 14 September 2022

Peer-Review Model

External peer-review was done through double-blind method.

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ABSTRACT

In this research, an extracted pectin from cocoa pod husks through Soxhlet extraction method using nitric acid as solvent. The optimization of the process was conducted using response Surface Methodology (RSM) and Artificial neural Network (ANN). The physicochemical properties of the pectin were carried out to determine its potential. In this research work, the Cocoa pod husks used were obtained from Osiakpu community in Ogba/Egbema/Ndoni Local Government Area of Rivers State and later transported to the chemical laboratory of Rivers State University, Port Harcourt, for experimental purposes. The husks were washed thoroughly, dried using the oven and then grounded into powder (flour). Pectin was then extracted from the cocoa pod husks flour with the use of Soxhlet extractor. A three-level-three-factor Box-Behnken design was considered and used; experimental runs were initiated of over 15 times using Response Surface Methodology (RSM). Some independent factors considered in this research process include cocoa pod husk flour weight (CPHFW), extracted time (min) and acidulated water pH (diluted nitric acid). Results showed that the optimum yield (11.62%) as predicted by RSM at the conditions: cocoa pod husk flour weight (CPHFW), 35.2175g; extraction time (ET), 73 min and acidulated water pH (ADWpH), 1.5 was in agreement with the experimented optimum yield (12.41%). The extracted dry pectin (11.04%) at these conditions has 67% methoxy content, >70% galacturonic acid content and 67% degree of methylation. This research hereby resolved that for an optimum yield of this process, certain factors such as ADWpH should be considered and extraction time. It was also observed that higher ADWpH and moderate extraction time for increases pectin yield.

Keywords: pectin, extraction process, response surface, optimization, artificial neural network, ripe cocoa, pod husks



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1. INTRODUCTION

Cocoa pod (*Theobroma cacao*) is a very pivotal agricultural and economic crop that is grown in large scale in so many tropical areas such as Ghana, Nigeria, Cameroon, Brazil, Ecuador, Columbia, Mexico, Indonesia, Malaysia etc [1].

When ripe cocoa pods are harvested from the trees and piled off in order to obtain the beans inside. After fermentation of surrounding pulp, the beans are allowed to dry and bagged, constituting the cocoa of commerce, used mainly by chocolate manufacturing companies [2]. During the extraction of cocoa fruit [3], are thrown away and may cause an environmental problem when dumped around the processing plants. Cocoa pod husk are also a significance source raw materials used for the cure some diseases especially the black pod root [4]. The burden of cocoa pod husks waste continues to increase and represent a serious challenge for waste management [5]. Total production of cocoa bean is estimated worldwide to be approximately 4.0 million tons in 2013, with a value of about \$12 billion [6]. Deposits from cocoa processing industries are many such as the hull, shell etc which contributes to the higher proportion rate of approximately 85%, whereby the annual worldwide amount of cocoa pod husks is estimated to be about 55 million tons, which is equal to 13 times the total amount of cocoa beans available [7]. Therefore, cocoa pod husks need to be exploited to produce high-value-added products [8]. The increase in industrial demand of pectin with differential aim to stabilize the products increases the need for pectins of different types or derivatives with tailored properties [9-10].

Research aim and objectives

1. To optimize the extraction of pectin from ripe cocoa pod husks using response surface methodology and artificial neural network
2. To extract pectin from ripe cocoa pod husks pod husks
3. To optimize the extraction in using artificial neural network and response surface methodology.
4. To characterize the physicochemical properties of determining the potential uses of (ii).

2. MATERIALS AND METHOD

This section of the research describes the materials and methodology that was used in this research work; ranging from setting of instrument for data collection, method of data collection, method of data analysis (which includes the materials used, equipment and reagents, preparation of the reagents, refining process and the characterization of the pectin.

Cocoa pod Husks (*Theobroma cacao* L.)

Dry cocoa pod husks were gotten from Osiakpu Cocoa Plantation and Processing in Obot Akara Local Government of Akwa Ibom, Nigeria.

Chemical Solvents

All chemicals and solvents such as ethanol, NaOH, HCL, nitric acid and more, used were within analytical reagent grade.

Glassware

The glassware used in this work include; include beakers, round-bottom flasks, conical flasks, Petri dish, measuring cylinder and burettes etc.

Equipment

The equipment used in this work is detailed below

Soxhlet Extractor

Soxhlet extractor of 500 ml was for the extraction of pectin from the cocoa pod husks flour.

Digital Weighing Balance

The digital weighing balance was used to determine all the weights of the extract, chemicals and other substances used throughout the duration of this research experiment.

Heating Mantle

The heating mantle was used as a source of heat to enable the heating of the solvent. And it was used in carrying out the physiochemical properties of the pectin where heating was necessary.

Oven

The oven was used to determine the moisture contents of the pectin. It was also to dry the cocoa pod husks. The oven has trays for drying, temperature control knob and overheated control buttons.

Centrifuge

The centrifuge was used to get a clear two phases of the pectin mixture.

CHARACTERIZATION OF THE OPTIMIZED PECTIN

The approximate yield of pectin was determined considering the weight of the pectin extracted when dried with an oven. See below;

$$\text{Yield of pectin (\%)} = \frac{\text{Pectin Extracted (g)}}{\text{Cocoa Pod Husks Flour (g)}}$$

Moisture Content

Pectin's moisture was obtained by weight different after oven drying 100 mg of dry pectin at 130°C for 1 hour.

Methoxy Content

This determines the functional group or properties of pectin solution and can also affect the structure and texture of pectin gel. According to [11], 0.1 of dry pectin was weighed into 100ml of distilled water 2 ml of 70% alcohol was added and then heated 5 drop of phenolphthalien was added and then titrated with 0.05N of NaOH. Equivalent point was recorded as V1. HCL (0.1M) of volume 20 ml was added to the brownish pink coloured solution and allowed to stand for 15 minutes. The solution was then shaken until the pink colour. The volume gotten was recorded as V2. Methoxy content was analysis from the equation below.

$$\text{Methoxy Content} = \frac{V2 \times N - NaOH \times 100}{\text{Pectin(g)} \times 1000}$$

3. RESULTS AND DISCUSSION

Experimental design was employed in the evaluation and determination of experimental values and results with the aid of design expert version 11.1.0.1 software in order to optimize the extraction process. The experiment was designed on three levels factors that generated 17 experimental runs. The three independent factors were cocoa pod husks flour weight, CPHF (g); extraction time, ET (min) and acidulated water pH. ADWpH (diluted nitric acid). It was observed that the predicted values for the extraction by RSM and ANN were close to the experimented values obtained from the laboratory, which made the residual non-significant. The pectin yield ranged from 5.83% (wet basis). The highest yield was obtained when the CPHF extraction conditions were CPHFW, 30g; Extraction time, 80 min and pH 4.0 with the aid of RSM. [10] optimized the extraction of pectin from cocoa pod husk using hydrochloric acid with different concentration of HCL (0.653, 1.5.2 and 2.282 M.) temperature (43.18, 50.60, 70 and 76.82°C) and extraction time (3.318, 4.5.6 and 6.282 hours). The highest obtained pectin yield was 12% (wet basis) and extraction time of 4.8 hours. In this work nitric extracted pectin reached a yield of 22.16% (wet basis). This pectin yield is a little higher than that obtained by [11] when they used lemon and orange peels (20.75% and 15.25% respectively; all wet basis).

Table 1: Build Information for Optimization of pectin Yield by RSM

File Version	11.1.0.1		
Study Type	Response Surface	Subtype	Randomized
Design Type	Box-Behnken	Runs	17
Design Model	Quadratic	Blocks	No blocks
Build Time (ms)	4.00		

Table 2: Model summary Statistics for RSM

Name	Units	Types	Changes	Std. Dev.	Low	High
CPFW		Factor	Easy	0	30	40
ET (min)		Factor	Easy	0	60	100
ADWpH		Factor	Easy	0	1.5	4
Pectin yield %(w/w)		Response		0.413499	5.83	22.16

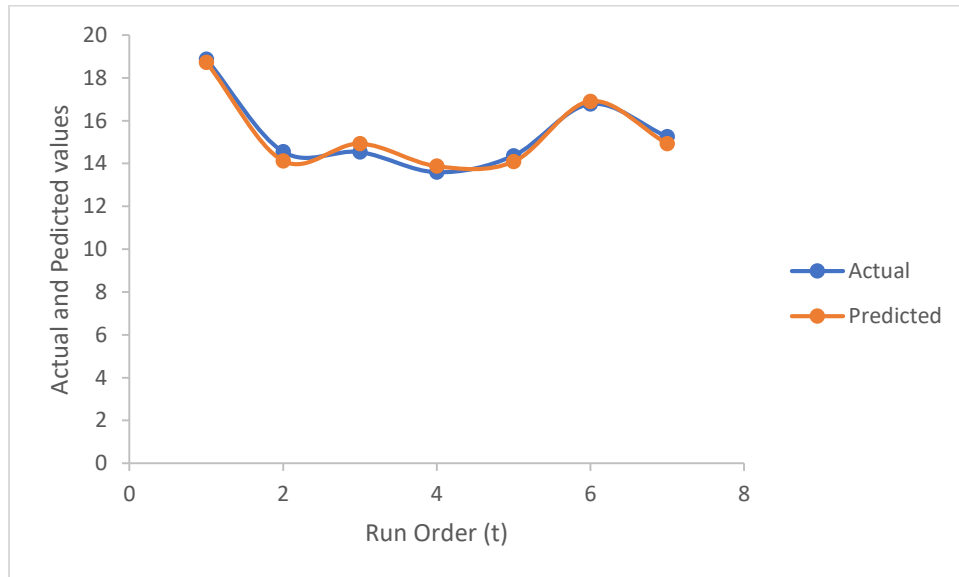
**Figure 1: Plot of Actual Experimental values and Predicted values against time**

Figure 1 shows the plot of actual experimental values and predicted values against time. The trend show similarity for both predicted and actual values. Though there is a slight different especially from run order 2 to run order 4.

**Figure 2: Plot of Actual Experimental values and residual (RSM) values against time**

Figure 2 illustrates a plot of actual experimental values and residual (RSM) values against time. However, in this case, the trend of the plot shows massive differential movement from the beginning of the plot to the end.

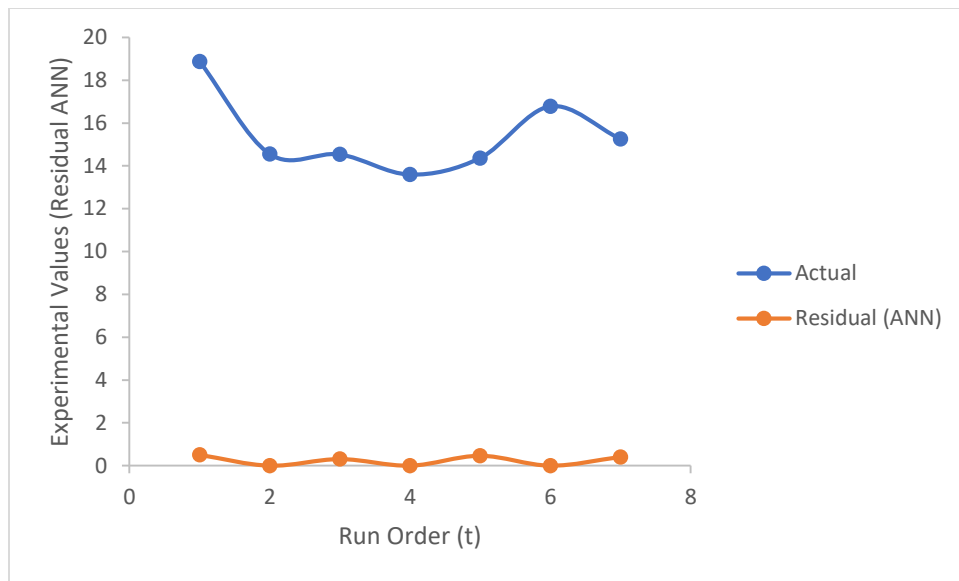


Figure 3: Plot of Actual Experimental values and residual (ANN) values against time

Figure 3 demonstrate a plot of actual experimental values and residual (ANN) values against time. However, in this case, the trend of the plot shows massive differential movement from the beginning of the plot to the end.

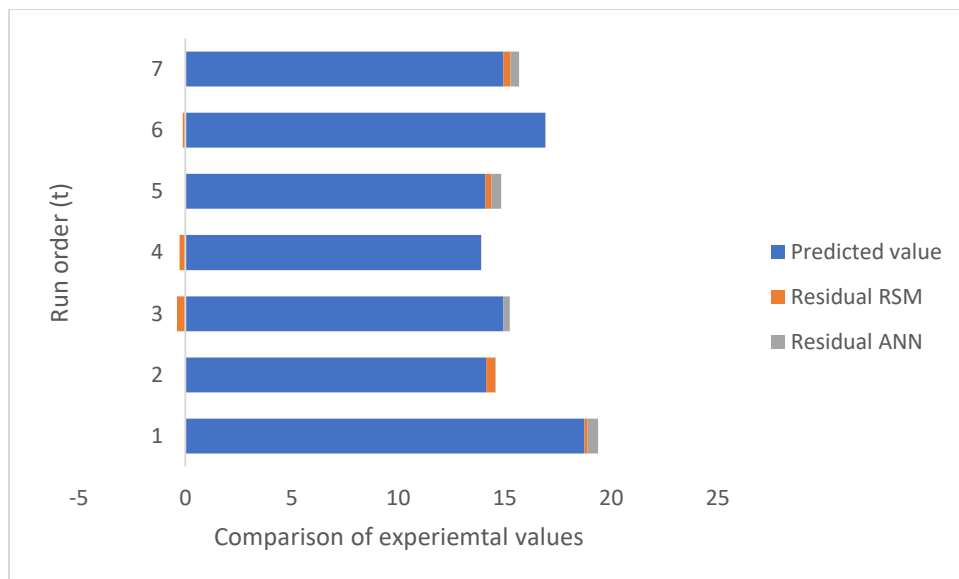


Figure 4: Plot of Comparison of predicted values, Residual values (RSM) and residual (ANN) values against time.

Figure 4 is a plot of Comparison for predicted values, Residual values (RSM) and residual (ANN) values against time. The plot shows a dominant state for the predicted values, keeping the residuals for both RSM and ANN in a close relationship.

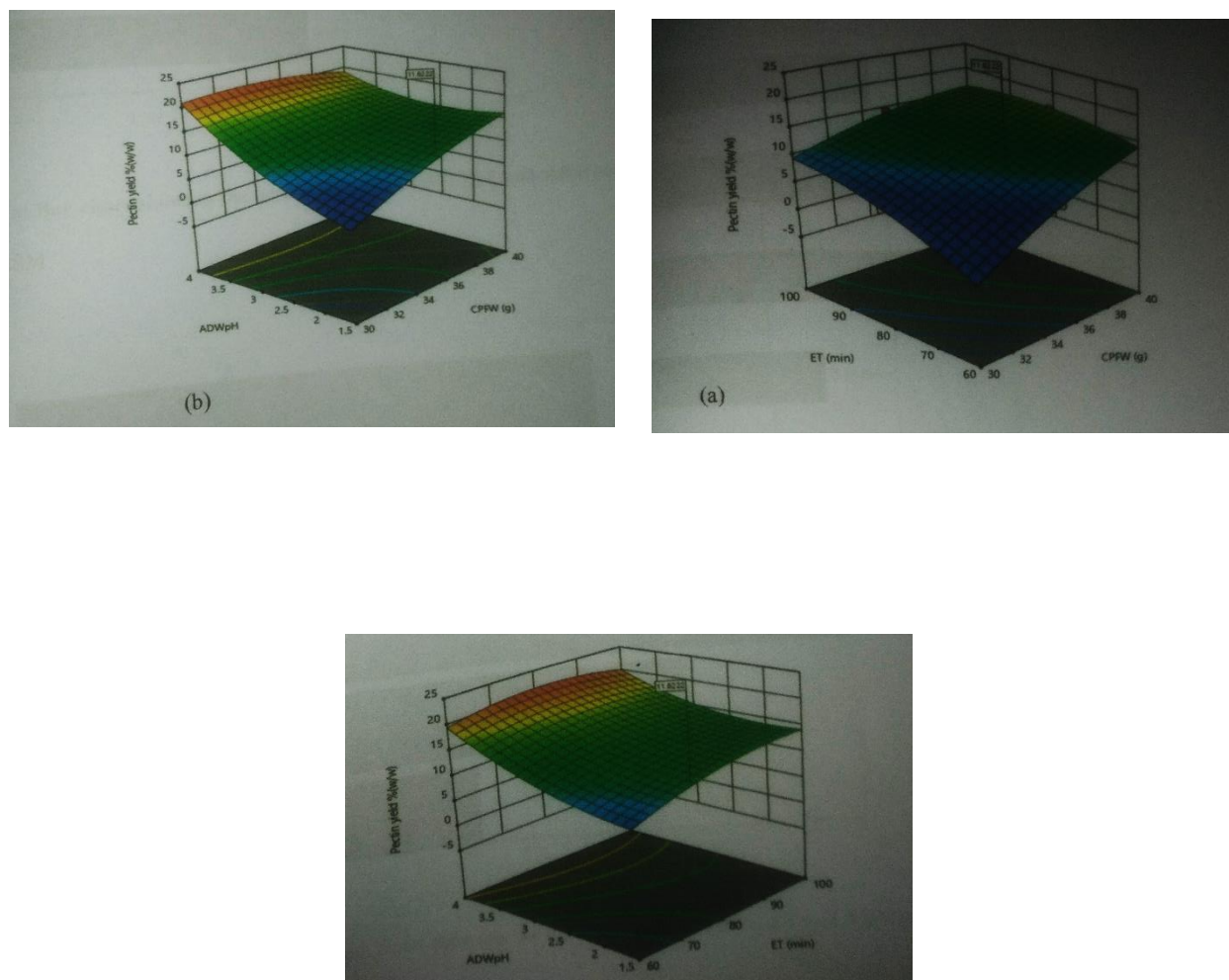


Figure 5: 3D Surface Plots showing the effects of the Three Factors on Pectin yield

Figure 5 shows the estimated effects of cocoa pod husk flour weight, CPHFW: extraction time, ET and acidulated water pH, ADWpH on the pectin yield. From 5a, it could be said that the higher the extraction time and CPHFW, the higher the pectin yield obtained and vice versa. The yield also increases significantly with increasing ADWpH and CPHFW as shown in Figure 5a. Also, the higher the ADWpH despite an increase in extraction time and CPHFW will lead to reduce or lower yield of pectin as seen in Figure 5b and 5c.

4. CONCLUSION

Dry beans produced in Nigeria generates approximately ten tons of cocoa pod husks which makes the burden of cocoa pod husks waste continue to increase and represents a serious challenge for waste management. As a way of remediating to ecological problems and adding value to them, the pectin extracted from cocoa pod husk were investigated and results interestingly showed that valuable pectin could be recovered from husks. The optimum pectin yield predicted by RSM (11.62% wet basis) and the experimented optimum yield (12.41 wet basis) gotten from the laboratory base on the optimized conditions (CPHFW, 35.2175g; extraction time, 73min and acidulated water pH. 1.5) were closely in agreement. The optimum dry pectin yield (11.04%) had methoxy content of 66.65% galacturonic acid content and 66.67% degree of methylation.

Funding

This study has not received any external funding.

Conflict of Interest

The author declares that there are no conflicts of interests.

Data and materials availability

All data associated with this study are present in the paper.

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